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Characterization of 'Balkan flint' artefacts from Bulgaria and the Iron Gates using LA-ICP-MS and EPMA

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АРХЕОЛОГИЧЕСКИ ИНСТИТУТ С МУЗЕЙ – БАН
ARCHAEOLOGICAL INSTITUTE AND MUSEUM – BAS

ИНТЕРДИСЦИПЛИНАРНИ
ИЗСЛЕДВАНИЯ
XXII - XXIII

INTERDISCIPLINARY STUDIES

CONTENTS

1. <i>Lazar Ninov</i> . Editorial.....	7
2. <i>Clive Bonsall, Maria Gurova, Chris Hayward, Chavdar Nachev, Nikolas Pearce</i> . Characterization of “Balkan flint” artefacts from Bulgaria and the Iron gates using LA-ICP-MS and EPMA.....	9
3. <i>Kuncho Kunchev</i> . Theoretical formulation of flint cleavage	19
4. <i>Martin Nikolov</i> . Petrographic analysis of stone shaft-hole axes from North Bulgaria Bronze Age	28
5. <i>Hristo Popov, Zdravko Cincov</i> . Preliminary archaeometallurgical data from the region of the villages of Valche Pole and Kamilski Dol, the Eastern Rhodopes.....	32
6. <i>Viktor Zaykov, Diana Gergova, P Khvorov, Plamen Bonev</i> . Archaeometric studies of Thracian golden objects from the National Archaeological Museum in Sofia	75
7. <i>Thilo Rehren, Anastasia Cholakova</i> . The Early Byzantine HIMT glass from Dichin, Northern Bulgaria	81
8. <i>Ralitsa Georgieva, Yanko Dimitriev, Bisserka Samuneva, Albena Detcheva</i> . Reproduction of medieval glasses found in Bulgaria using technical raw materials.....	97
9. <i>Bissera Tomova</i> . Textiles and woven materials in the IX – XVIII c. written sources current in the today’s Bulgarian territories (technology, production, processing and terms)	103
10. <i>Alexandra Buzhilova</i> . Palaeopathological observation of Neanderthal Skeleton from Kiik-Koba, Crimea.....	177
11. <i>Viktoria Russeva</i> . Distribution of enamel hypoplasia – new perspectives for interpretation of the anthropological material from archaeological sites in Bulgaria (preliminary results)	186
12. <i>Lazar Ninov</i> . The fauna of the Thracian town Helis (preliminary data).....	193
13. <i>Tzvetana Popova</i> . Application of the contextual and taphonomic analysis in the study of bio remains.....	201
14. <i>Tsoni Tsonev</i> . Information technologies and archaeological heritage of Bulgaria	206

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**ИНТЕРДИСЦИПЛИНАРНИ
ИЗСЛЕДВАНИЯ**

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CHARACTERIZATION OF ‘BALKAN FLINT’ ARTEFACTS FROM BULGARIA AND THE IRON GATES USING LA-ICP-MS AND EPMA

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Introduction

In 2009 at the 15th annual meeting of the EAA in Riva del Garda, Italy, a special session was organized on *Balkan Flint in Southeast European Prehistory*⁶, which brought together scholars whose research on the early farming societies of South-east Europe had inevitably led them to confront the problem of the appearance at the beginning of the Neolithic of a new, high-quality, raw material for the manufacture of chipped stone artefacts, widely known as ‘Balkan Flint’ or yellow spotted flint, and its continued use in some areas into later periods. How and where was it obtained, and why was it so popular? Did it hold symbolic as well as economic and technological significance for Neolithic peoples?

Since the 1970s it has been the conventional view that this distinctive flint originated from a source or sources on the so-called ‘Pre-Balkan Platform’ (Moesian Platform) in northern Bulgaria from where it was distributed throughout much of South-east Europe (Kozłowski & Kozłowski 1984, Voytek 1987). But did it all come from sources on the Moesian platform and, if so, where were those sources located? The present paper is concerned with the very important but still unresolved problem of the provenance of Balkan Flint, and the preliminary re-

sults of trace element analyses using LA-ICP-MS and EPMA techniques are discussed.

Archaeological background

The ‘Balkan flint’ problem emerged from Bulgarian prehistoric evidence and subsequently became deeply embedded in Balkan research, especially that concerned with the supra-regional Karanovo I-Starčevo-Criș-Körös cultural complex.

Inherently linked to the Neolithisation debate, the ‘Balkan flint’ problem remains intractable. In spite of decades of research on the origins and spread of the Neolithic in the Balkans, it has proved difficult to explain the appearance of standardized (formal) toolkits made of yellowish-brown, white-spotted ‘Balkan flint’ ca 6000 BC. Was the concept of the toolkits brought with migrants along currently unknown routes from some part of (central or north-western) Anatolia, or did it originate in the context of local pre-Karanovo enclaves? Two potential ‘nuclear areas’ for the development of the technological and stylistic features of these toolkits have been suggested by M. Gurova (Гюрова 2009, Gurova 2008): 1) the region of the Struma and Vardar valleys, which some authors believe were directly and independently colonized by Anatolian migrants (Николов 1987, Lichardus-Itten 1993, Lichardus-Itten et al. 2006), and 2) the area in northern Bulgaria where the earliest Neolithic sites are believed to be those characterized by ‘monochrome pottery’, whose inhabitants were already experienced in blade production and lived in reasonable proximity to the abundant high-quality flint outcrops of the Lugorie region to the east (see also Тодорова, Байков 1993).

Gurova (2008) has argued convincingly that Bulgarian Early Neolithic chipped stone assemblages contain coherent and diagnostic formal flint toolkits across the vast Karanovo I–II culture area.

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⁶ The session was organized by Maria Gurova (Bulgaria), with co-organizers Clive Bonsall (UK), Barbara Voytek (USA), and Dušan Borić (Serbia/UK)

The toolkits are characterized by long, regular blades with (bi) lateral, semi-abrupt, high retouch and sometimes with rounded or pointed ends, as well as highly (re-) used sickle inserts, all made of high-quality yellowish-brown or honey-coloured flint with sporadic whitish spots (usually referred to in the literature as ‘Pre-Balkan Platform flint’, or simply ‘Balkan flint’) (Fig. 1). One of the most challenging questions regarding these toolkits is the identification of their raw material outcrops, supply strategy, and the network of their widespread distribution (local and supra-regional). The spatial distribution of tools made from this distinctive raw material is very extensive: artefacts made of ‘exotic’ Balkan flint have been reported from Neolithic Greece, Serbia (including Vojvodina), Romania, Macedonia, and Hungary (for details, see Gurova 2008).

Standardized toolkits of Balkan flint were abundant throughout the “classical” Early Neolithic Karanovo I and II periods of the Tell Karanovo sequence, *ca* 6000 to 5500 cal BC.⁷ Since they continue to be found until the end of the Karanovo III period at Tell Karanovo, *ca* 5500–5280 cal BC (Görsdorf 1997, 379), this can be regarded as a *terminus ante quem* for the presence of formal toolkits.

Only a few sites in Bulgaria offer the possibility of studying the formal tools over an extended period of time and in a variety of contexts; these are Karanovo, Slatina, and Yabalkovo (cf. Fig. 3). Although an impressive corpus of flint studies has been generated over the past two decades, many questions regarding these flint toolkits remain – what was their technical origin, where were the outcrops from which the raw material was obtained, what was the system of procurement and distribution, who were the manufacturers (flint knappers), where were their workshops, and what was the nature and result of their interactions with neighbouring Early Neolithic cultural groups and identities?

Raw material outcrops of the Balkan flint

It is worth mentioning the state of the knowledge of this topic within Bulgarian archaeology. There have been occasional studies of cryptocryst-

alline siliceous rocks (‘flint’) over the past three decades. The first to highlight the abundance and variety of the flint sources in north-east Bulgaria was K. Kanchev, who attempted to construct a database and to link the flint outcrops identified with prehistoric artefacts and their circulation (Кънчев 1978, Кънчев и др. 1981).

Ivan Gatsov presumed north-west Bulgaria to be the region of provenance of the raw material used for the Early Neolithic assemblages from western Bulgaria (Gatsov 1993, 40–41). In the same publication the Russian specialist, Natalia Skakun, noticed that “certain specimens are probably made of Dobrudzha flint” (Skakun 1993, 54).

Two principal types of flint were recognized among the assemblages from the tells of Karanovo and Azmak, that referred to as ‘type A’ corresponding to Balkan Flint. The work was done by Ivan Gatsov and the geologist, Kurčatov, who suggested the abundance of artefacts was due to the proximity of local flint outcrops, and identified outcrops (more theoretically than actually) in the region of the Sveti Ilia hills in eastern Thrace, not far from the tells (Gatsov and Kurčatov 1997, 215). This interpretation has been quoted repeatedly, but not substantiated by further systematic research. Subsequently, various attempts at Balkan Flint provenancing have been made based on visual inspection and macroscopic comparisons.

Preliminary examination of a series of artefacts from Yabalkovo led Zlateva-Uzunova to conclude that “...the predominant raw material with identified origin comes from deposits in the Upper Thrace, the Sredna Gora, north [read western] Bulgaria and the eastern Rhodopes” (Leshtakov et al., 2007, 201). The same author described two types of flint raw material from Ohoden (beige-wax, and yellow with whitish spots) thought to originate from the Dobrudzha region (Zlateva-Uzunova 2009, 70–72). It is worth noting that these flint types appear identical to the raw material used at the Yabalkovo site, discussed above.

In fact, apart from some conjecturing by Natalia Skakun, the first to presume (albeit theoretically) a northeastern provenance of the raw material used for the Neolithic big blades was T. Tsonev, in the context of his theory about the role of long blades in the “communal perception of long distance exchange through common metaphors” (Tsonev 2004, 262).

It was against this background of disagreement about the provenance of Balkan Flint, and a desire

⁷ This age range is based on the 14C evidence from the sites of Azmak, Eleshnitsa, Chavdar, Kovačevo, Slatina, and Galabnik (cf. Boyadziev 1995, 179–181, Lichardus-Itten et al. 2006, Thisen 2000)

to approach the problem systematically, that small-scale research collaboration between the archaeologist, Maria Gurova, and the mineralogist, Chavdar Nachev, was established.

Sedimentological aspects of Balkan Flint

Based on extensive research into the occurrence of siliceous rocks and their prehistoric use, Ch. Nachev has recognized four distinct types of flint in Bulgaria: Hemus flint, Dobrudzha flint, Moesian flint, and Rhodope flint (Кънчев и др. 1981, Начев 2009, Nachev and Kanchev 1984). Each type has a different geographical distribution, geological age, and diagnostic features (see Gurova and Nachev, 2008, fig. 5). The mineralogical comparison of these four types distinguishes Dobrudzha flint as the most suitable material for knapping — the unique homogeneity and the size of the nodules permitting core preparation and debitage of large laminar blanks.

Archaeologically, the most significant accumulations of siliceous concretions occur on the Moesian Platform and adjacent parts of the Balkan Alpine Orogen. The main lithostratigraphic horizons in which these occur are of Lower Cretaceous (Aptian) and Upper Cretaceous (Coniacian, Campanian and Maastrichtian) age. Both series are represented on the Moesian Platform in northern Bulgaria. Of lesser archaeological significance were Upper Jurassic (Oxfordian) flint, termed Hemus flint, the hydrothermal chalcedony veins of the Upper Cretaceous Sredna Gora zone (Sredna Gora atypical flint), and the flints of the Oligocene Rhodopes volcanic zone (Rhodopes atypical flint). In the broadest sense of the term, 'Balkan flint' can be taken to mean every flint type on the Moesian Platform and adjacent parts of the Balkan Alpine Orogen, including both Lower Cretaceous (Aptian) and Upper Cretaceous (Campanian and Maastrichtian) flint.

The following is a short description of the two main types of flint found on the Moesian Platform (for further details, see in Начев 2009, Gurova and Nachev 2008):

Ludogorie or (Dobrogea) flint. The silica concretions are hosted in Lower Cretaceous (Aptian) micrite limestones. The Aptian flint-rich limestones are the source for various types of flint found in secondary (placer) deposits. The majority of these are eluvium–proluvium deposits, where angular pieces of flint occur in soft sandy-carbonated masses.

Examples include Kriva Reka, Tetovo, Kamenovo, Ravno, and Chukata (near Razgrad). Other placer deposits in the region are of palaeoalluvial type, such as the Drianovets locality. Aptian flint has a wide geographical distribution in northeast Bulgaria, to the north of Novi Pazar and between Rouse and Dobrich. The main outcrops are near Vetovo, Koubrat, Razgrad, Isperih, and Novi Pazar. Two types of Ludogorie flint can be distinguished microscopically:

(i) The *Ravno type* is found in the northwest of the area, along the Topchii River near Topchii, Kamenovo, Ravno, Koubrat, Belovets, Tetovo, and Chereshovo. It is characterized by a micro- to crypto-crystalline groundmass and individual sponge spiculae.

(ii) The *Kriva Reka type* occurs in the southeast part of the area — between the villages of Goliem Porovets, Drianovetsa, Krivnia, Chukata (Razgrad), Lisi Vrah, Kriva Reka and Rouzhitsa. Its microscopic characteristics consist of microcrystalline aggregates with recrystallization of chalcedony.

Moesian flint. The silica concretions are hosted in the Upper Cretaceous (Campanian) chalk, chalk-like limestones, and fine grained biomorphic limestones (Maastrichtian). The Upper Cretaceous flint-rich rocks form three large outcrop areas in northern Bulgaria (the Moesian Platform and adjacent parts of the Balkan Alpine Orogen) from west to east as follows: the first between Montana and Lovech, the second between Pleven and Nikopol, and the third between Shumen and Devnya.

Within this large territory Moesian flint has a broad distribution and has formed extensive deposits. It displays similar features throughout the entire area. This and the convenient transport route along the Danube suggest the major outcrops on the Danube bank near Nikopol and Somovit could be the source of flint raw materials found at archaeological sites over a very broad area, including parts of Serbia and Romania. Microscopic examination shows abundant siliceous biogenetic relicts (fragmented and chaotically distributed microfauna).

A small series of archaeological samples was submitted to Chavdar Nachev for raw material identification by comparative thin-section analysis with flint from known sources across the Moesian Platform. Three samples taken from the Dzhaulunitsa, Rakitovo and Yabalkovo sites show typical crypto-crystalline structure and microfaunal remains (Fig. 2). Subsequently, samples from three other Bul-

garian Early Neolithic sites (Slatina, Ohoden, and Kovačevo), as well as from the Early Neolithic site of Aria Babi in the Serbian Iron Gates area, were included in the study. Unfortunately, thin section analysis proved to be of limited value in discriminating between the samples, permitting only the following, very general, conclusions:

- No reliable identification of raw material type was possible for the archaeological samples;
- The archaeological flint samples from Rakitovo, Slatina and Ohoden are probably derived from the ‘Moesian’ flint region;
- None of the archaeological samples examined could be related with confidence to the ‘Ludogorie’ raw material outcrops, while the Kovačevo flint does not correspond to any known raw material type.

Trace-element Analyses

The somewhat inconclusive results from comparative thin-section analyses led us to consider other means of identifying the source or sources (*characterization* or *provenancing*) of the ‘Balkan Flint’ used by Early Neolithic communities in Bulgaria and neighbouring regions of Southeast Europe, and by extension the exchange networks within which it

was distributed.

Flint is composed predominantly of silicon (Si) and oxygen (O), and the concentration of these two major elements tends to be broadly similar whatever the source. However, the *trace elements* in flint (elements present in very small quantities, i. e. at average concentrations of less than a hundred parts per million) do vary between sources. Therefore, through trace-element analysis it may be possible to discriminate between Balkan Flint sources and to assign artefacts to sources.

In a pilot study to test the effectiveness of trace-element analysis as a tool for characterizing Balkan Flint, we used *laser ablation inductively coupled plasma mass spectrometry* (LA-ICP-MS) and *electron probe micro analysis* (EPMA) to investigate the chemical compositions of flint samples from a number of geological sources and archaeological sites. Both techniques are capable of high precision quantitative chemical analysis with high spatial resolution (up to 1 micron and 25 microns for EPMA and LA-ICP-MS, respectively — for brief explanations of the techniques, see Reed (1995) and Perkins and Pearce (1995). The EPMA analyses were performed at the University of Edinburgh by Dr Chris Hayward, and the LA-ICP-MS analyses at the University of Wales by Dr Nick Pearce. EPMA analyses were

Table 1.

Details of flint samples submitted for trace element analysis (site locations are shown in Figure 3)

Site	Sample I. D.	Context	Collected
Mramoren	1	Balkan Orogen – K2 Campanian	
Somovit	2	Moesian Platform – K2 Campanian	
Nikopol	3	Moesian Platform – K2 Campanian	
Mouselievo	4	Moesian Platform – K2 Campanian	
Ravno	5	Moesian Platform – K1 Aptian	
Drianovets	6	Moesian Platform – K1 Aptian	
Goliam Porovets	7	Moesian Platform – K1 Aptian	
Kriva Reka	8	Moesian Platform – K1 Aptian	
Shumen	9	Moesian Platform – K2 Campanian	
Kovačevo	Kv-1	Early Neolithic – Sq. G, niveau 6, sac 55238	2001
Kovačevo	Kv-2	Early Neolithic – Sq. F, niveau 3, sac 14227	1989
Ohoden	O	Early Neolithic – I 19	2008
Slatina	S	Early Neolithic – I hab. hor, C 450	1986-8
Dzhuljunitsa	D-1	Early Neolithic – Trench XIII, N 15	2005
Dzhuljunitsa	D-2	Early Neolithic – Trench XIII, N 748	2005
Yabalkovo	Y-1	Early Neolithic – Sq. K 38	2003
Yabalkovo	Y-2	Early Neolithic – Sq. G22	2003
Schela Cladovei	SC-1	Early Neolithic – Sq. 5, AIII VI. 1, N 21	1992
Schela Cladovei	SC-2	Early Neolithic – Sq. 7, AIII VI. 6, N 706	1993
Aria Babi	AB	Early Neolithic – surface collection	2005

performed using a Cameca SX100 electron probe microanalyser operated at 25 kV and 200nA with 1 μm beam diameter. Trace element analysis by LA-ICP-MS was performed using a Coherent GeoLas 2 Excimer laser ablation system operating at 193 μm coupled to a Thermo Element 2 sector field ICP-MS following methods described in Pearce et al. (2004). Ablation craters were 20 μm in diameter and average SiO_2 concentrations (from EPMA) were used for calibration of the analyses against the NIST 612 reference glass (Pearce et al. 1997). Analyses are the averages of three acquisitions from each flint.

Nine geological sources were tested. Single samples were analysed from four sources in the Lower Cretaceous (K1) rocks of the Moesian Platform in northeast Bulgaria (Fig. 3, sites 5-8) and from five sources in Upper Cretaceous (K2) formations of the Moesian Platform and the Stara Planina (Fig. 3, sites 1-4 and 9). Artefacts from seven Early Neolithic sites were also included in the pilot study (Fig. 4; Table 1). These comprise the sites of Dzhuljunitsa, Kovačevo, Ohoden, Slatina, and Yabalkovo in Bulgaria, and two sites in the Iron Gates section of the Lower Danube valley — Aria Babi in Serbia, and Schela Cladovei in Romania. Single samples were analysed from Ohoden, Slatina, and Aria Babi, and two artefacts from each of the other sites. Data were acquired for 24 separate elements (excluding Si and O) using EPMA and ICP-MS, although at the time of writing a complete set of results is available for only 17 elements.

Cluster analysis of the data for the archaeological samples reveals three 'groups' (Fig. 5), confirmed by the triangular plot of B-Ba-Pb (Fig. 6): (i) the samples from Aria Babi, Ohoden, Schela Cladovei, Slatina, Yabalkovo, and one of the samples from Kovačevo (Kv-2) form a single cluster with similar concentrations of all 17 elements; (ii) the two artefacts from Dzhuljunitsa cluster together, with relatively high Ba and low U; and (iii) one of the two artefacts from Kovačevo (Kv-1) forms a distinct chemical type with unusually high concentrations of 14 of the 17 elements, especially Ba, Ce, La, Nd, Pb, and Zr.

When the macroscopic characteristics (colour, 'spot' size and density) of the flint are taken into account, the geological samples from Mramoren (1), Drianovets (6), Goliam Porovets (7) and Kriva Reka

(8) appear distinct from the archaeological 'Balkan Flint' samples included in our pilot study, although it should be emphasized that macroscopic traits can vary within sources or even individual nodules. The remaining five geological samples would likely be classified as 'Balkan Flint' if found in an archaeological context. Of these, Nikopol (3), Mouselievo (4), Ravno (5) most closely resemble the archaeological specimens.

When the trace element data are considered, Nikopol stands apart from the archaeological samples having relatively high La and low B. Mouselievo and Ravno are similar geochemically to the main cluster of archaeological samples, although the Ravno sample has significantly higher Ba than any of the archaeological specimens. It is also worth noting that, although the samples from Kriva Reka and Somovit are similar geochemically to the two archaeological specimens from Dzhuljunitsa, they are distinct macroscopically, and no geological sample resembles Kovačevo-1.

Conclusions

A pilot study involving trace-element analysis of 'Balkan Flint' artefacts from Early Neolithic sites in Bulgaria and the Iron Gates section of the Danube Valley, and their comparison with geological samples from flint sources in northern Bulgaria, leads to three tentative conclusions:

- The archaeological 'Balkan Flint' samples are unlikely to be from a single source;
- The two artefacts from Kovačevo in southwest Bulgaria almost certainly came from different geological sources, and one of the artefacts differs geochemically from any of the north Bulgarian sources analysed;
- The majority of the geological samples analysed differ macroscopically and/or geochemically from the Neolithic 'Balkan Flint' artefacts included in the pilot study.

The results suggest that successful provenancing of flint artefacts is unlikely to be achieved through chemical analysis alone, and future research on the 'Balkan Flint problem' will doubtless require the use of other techniques, such as petrographic and micropalaeontological analyses, in combination with trace element studies.

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ХАРАКТЕРИСТИКА НА АРТЕФАКТИ ОТ „БАЛКАНСКИ” ФЛИНТ/КРЕМЪК ОТ БЪЛГАРИЯ И ЖЕЛЕЗНИ ВРАТА С ПОМОЩТА НА ЛАЗЕРНА АБЛАЦИЯ С МАССПЕКТРОМЕТРИЯ В ИНДУКТИВНО СВЪРЗАНА ПЛАЗМА И ЕЛЕКТРОНЕН МИКРОАНАЛИЗ

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(Резюме)

Характерна особеност на материалната култура през неолита в България и някои съседни райони е използването на висококачествен восьчно-жълт кремък с бели петнисти включения, известен в литературата като “балкански флинт/кремък”. Той е чест, а в някои случаи и неизменен атрибут на раннеолитните култури и културни комплекси с рисувана керамика на Балканите. Предложената статия съдържа първите резултати от проучване предприето с цел да тества конвенционалното и отдавна битово схващане, че всички артефакти от балкански флинт от неолитните селища в Югоизточна Европа произхождат от едно, или ограничен брой местонаходища от Северна България (т. нар. Предбалканска платформа), откъдето са разпространявани в различни части на Балканския полуостров.

Първата част на статията представя развитието на идеята за балканския флинт като исконно и контекстуално свързан с развитието на проучванията върху неолитните култури в България. Предложено е и кратко експозе на основните местонаходища на кремък в България с акцент върху тези от Мизийската платформа. Формулиран е изводът, че конвенционалните методи на анализ (микроскопско на-

блюдение с описание и сравнение на дюншлифи) не може да отговори на въпроса за идентифицирането и локализирането на източника/източниците на тази търсена и предпочитана през неолита суровина.

Финалната част на статията е посветена на резултатите от пилотно проучване, което чрез химически методи цели да установи произхода на кремъчни артефакти от някои добре познати и значими неолитни селища. Този подход е приложен към кремъчни образци от 9 геологически местонаходища на кремък в Северна България и 7 неолитни селища от България, Румъния и Сърбия, които са анализирани с използването на методите на LA-ICP-MS и EPMA (лазерна аблация с масспектрометрия в индуктивно свързана плазма и електронен микроанализ). Резултатите насочват към следните заключения:

- 1/ Балканският флинт използван от неолитното население по нашите земи със сигурност не произхожда от един единствен източник/местонаходище;
- 2/ Неолитните обитатели на Ковачево в Югозападна България са добивали своя “балкански флинт” най-малко от два химически разграничими и различни източника.



Fig. 1. Artefacts of 'Balkan Flint' from Yabalkovo (Early Neolithic site, 2003 excavation). Photo – M. Gurova

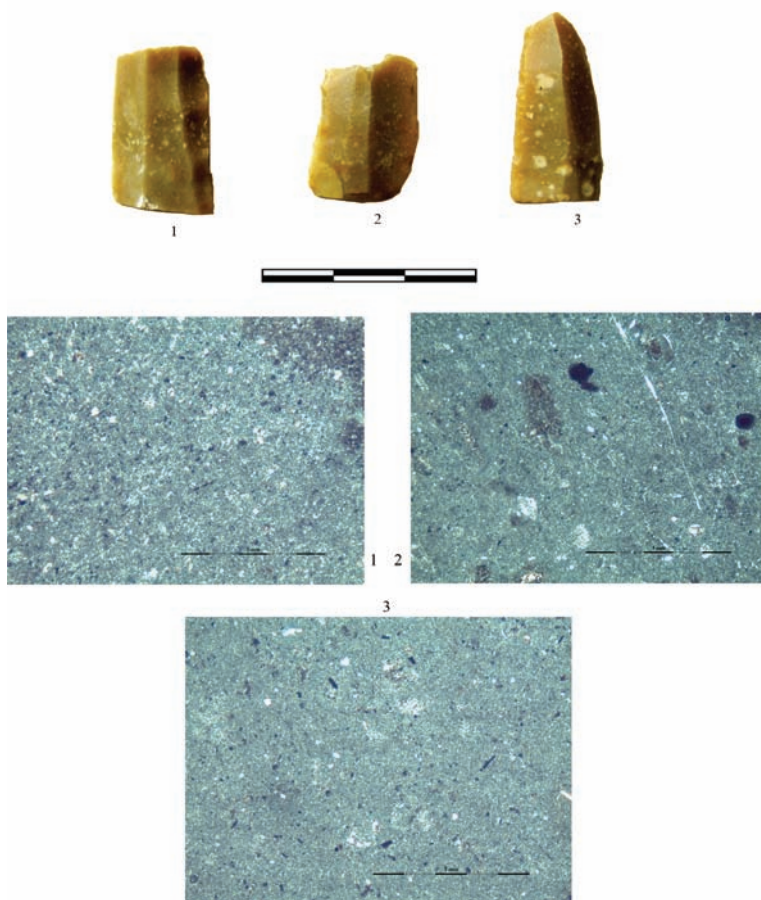


Fig. 2. Archaeological samples from the Early Neolithic sites: 1 – Dzuljunitsa, 2 – Rakitovo; 3 – Yabalkovo and thin-section photomicrographs corresponding to the respective site numbers (photo of artefacts – M. Gurova; photomicrographs – Ch. Nachev)

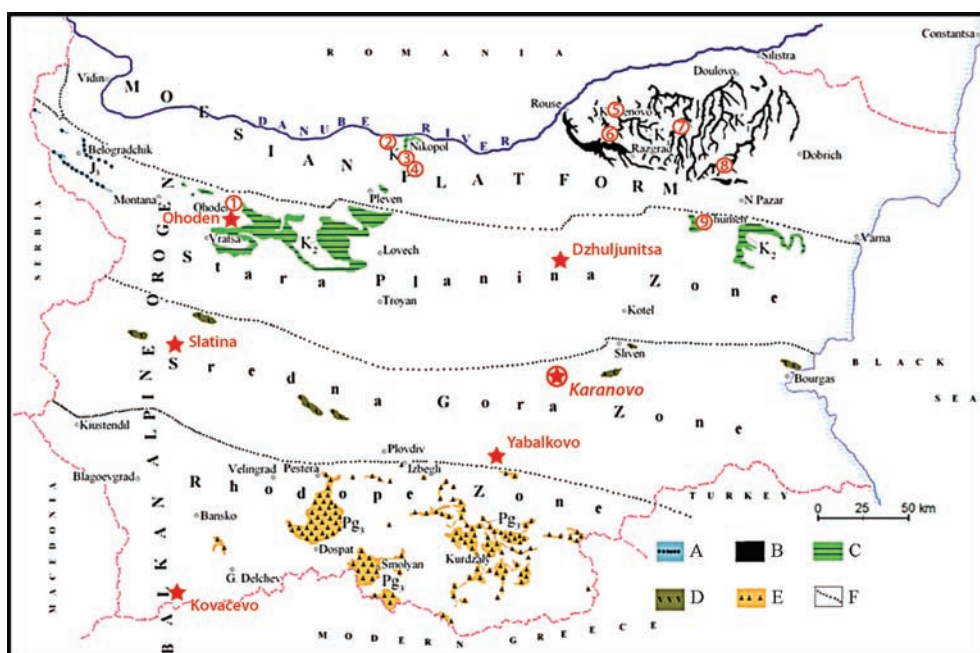


Fig. 3. Map of flint-bearing deposits in Bulgaria, showing the locations of geological (with numbers) and archaeological (with stars) samples included in the pilot study (cf. Table 1). Flint sources analysed: 1 – Mramoren, 2 – Somovit, 3 – Nikopol, 4 – Mouselievo, 5 – Ravno, 6 – Dri-anovets, 7 – Goliam Porovets, 8 – Kriva reka, 9 – Shumen. Geology: A – Upper Jurassic limestones, B – Lower Cretaceous (K_1) limestones, C – Upper Cretaceous (K_2) chalk and chalk-like limestones, D – Upper Cretaceous volcanic rocks with chalcedony veins, E – Oligocene volcanic rocks with chalcedony veins, F – Boundary between tectonic zones

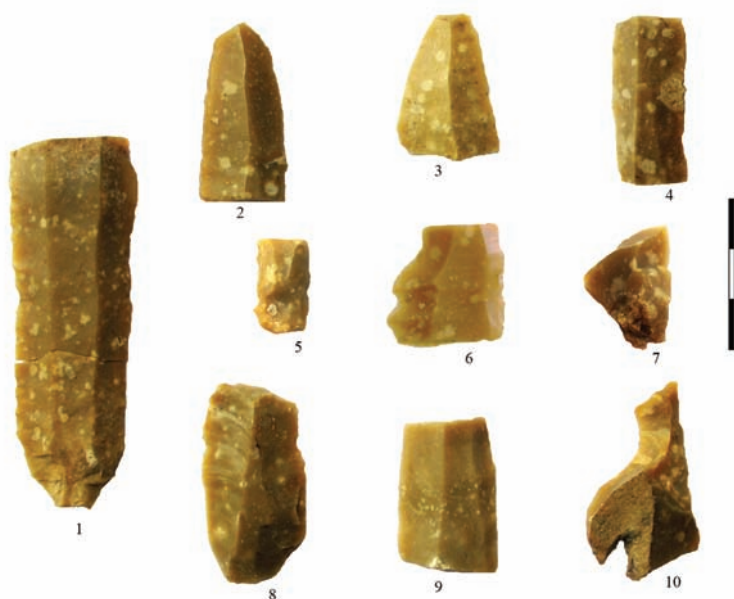


Fig. 4. Archaeological samples from Early Neolithic sites submitted to LA-ICP-MS analysis (Bulgarian sites 1–6 and 8–9). 1 – Slatina; 2 – Yabalkovo; 3 – Yabalkovo; 4 – Ohoden; 5 – Kovačevo; 6 – Kovačevo; 7 – Schela Cladovei (Romania); 8 – Dzhuljunitsa; 9 – Dzhuljunitsa; 10 – Aria Babi (Serbia). Photo – M. Gurova

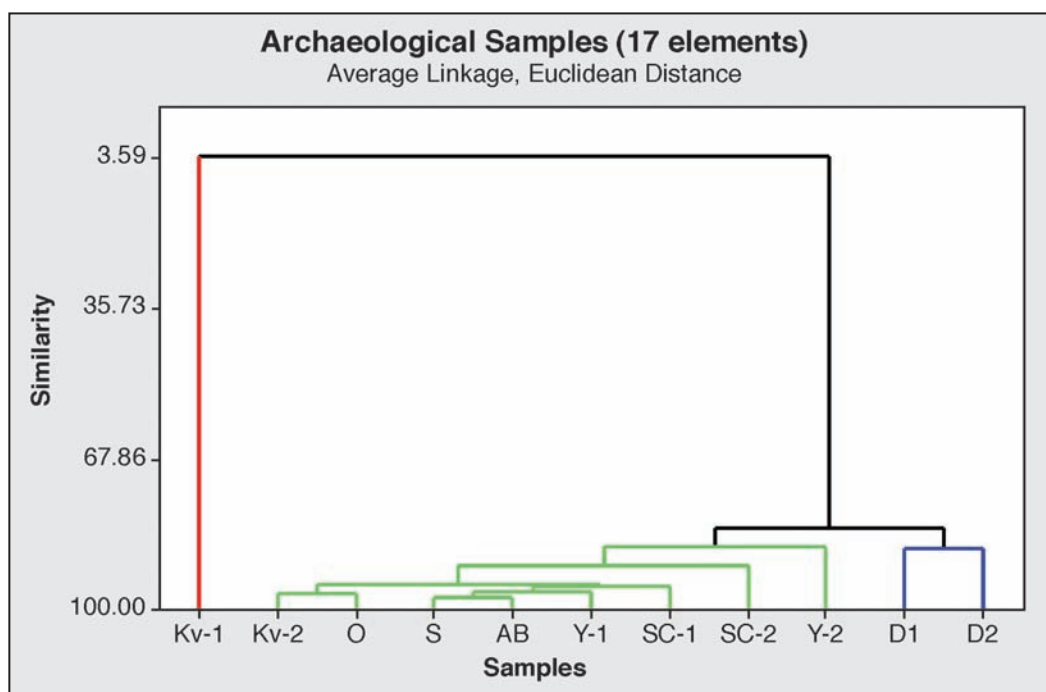


Fig. 5. Early Neolithic 'Balkan Flint' artefacts – cluster analysis using 17 elements determined by LA-ICP-MS. AB - Aria Babi, D - Dzhuljunitsa, Kv - Kovačevo, O - Ohoden, SC - Schela Cladovei, S - Slatina, Y - Yabalkovo

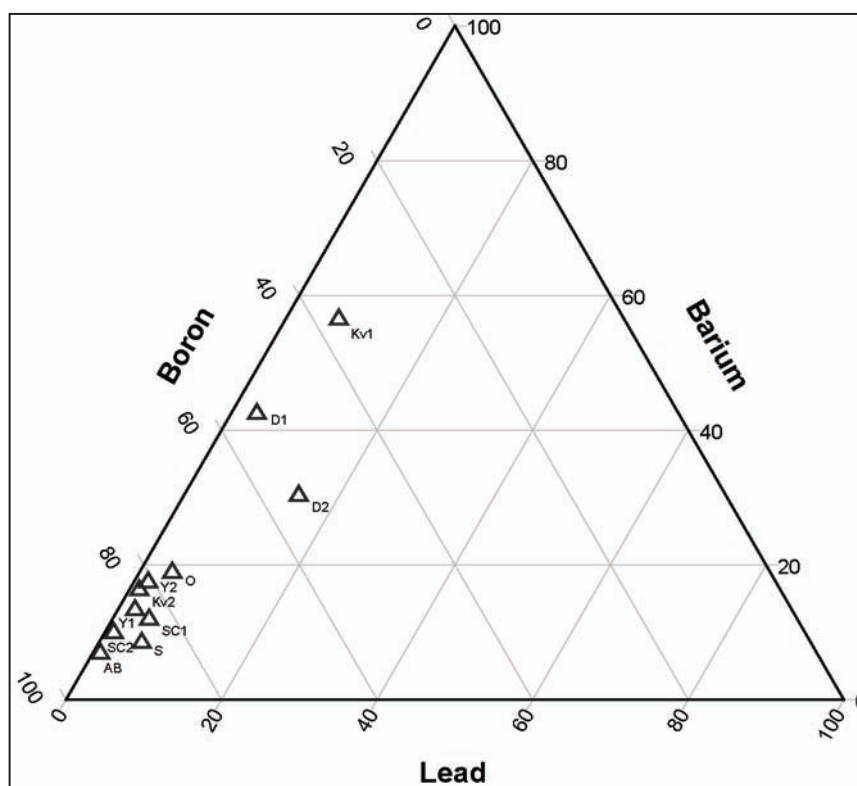


Fig. 6. Early Neolithic 'Balkan Flint' artefacts – trace element discrimination diagram using B, Ba and Pb determined by LA-ICP-MS: AB - Aria Babi, D - Dzhuljunitsa, Kv - Kovačevo, O - Ohoden, SC - Schela Cladovei, S - Slatina, Y - Yabalkovo